James Edward Sinclair

# THE EFFECT OF EXPLOSIVE MIXTURES UPON IMPACT SENSITIVITY

# UNITED STATES
# NAVAL POSTGRADUATE SCHOOL

## THE EFFECT OF EXPLOSIVE MIXTURES UPON IMPACT SENSITIVITY

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A Report
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Office of Naval Research
Upon an investigation conducted under
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TECHNICAL REPORT NO. 16

THE EFFECT OF EXPLOSIVE MIXTURES

Upon

IMPACT SENSITIVITY

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The discussions of the statistical nature of the problem with Professor C.A. Magwire of the Mathematics Department were also very helpful.

# ABSTRACT

The measurement of the impact sensitivity of two explosive systems TN~~B~~T-TNB and TNT-PETN has been studied along with relevant factors affecting this sensitivity. The effects of sample size, dilution with inert material, and active mixtures have been explored and a "rule-of-thumb" formula suggested as an index of the reliability of a particular explosive or explosive mixture.

# INTRODUCTION

The impact sensitivity of an explosive is usually considered today in the same manner as in the earliest experiments, namely, "The explosive must be as insensitive as possible and still explode when and where desired". This is not difficult since explosives such as trinitrotoluene (TNT) and trinitrobenzene (TNB) are available, which are extremely difficult to initiate without the aid of an explosive train or blasting cap and yet are easily "set off" with these aids.

The difficulty encountered in using explosives is that a certain number of explosions, due to impact alone, occur without normal cause. Many examples of unusual explosions occurred during World War II. In one incident, a 500 pound bomb loaded with TNT, but without the fuse or initiator attached, was dropped approximately one foot and exploded. Normally, one would expect such a bomb to be completely insensitive to this type of shock. In fact, many similar bombs did not explode even with fuses and initiators, when dropped in bombing raids. This property of explosives is well known and in evaluating impact sensitivity, one must take this unreliability into account since it exists in all explosives and no method has been found to eliminate it.

The most logical approach to a problem of this kind lies in the realm of statistics. The probability of an explosion occurring under strictly controlled conditions may be expressed by a statistical number obtained by a large sampling of data.

Under these conditions the pertinent point is not where the explosion occurs all the time or where it never occurs, but at some selected value, say fifty per cent of the time. This concept provides for a logical comparison of different impact sensitivities.

Therefore, if a machine can be set up to impact successive samples of an explosive, with an infinite number of varying impacts, over an infinite number of samples, this answer would be forthcoming. This method is impractical because the information gained is not proportional to the effort expended.

One method which has been used in practice, is to drop a weight on an explosive sample from one height for 100 times. This is repeated at different heights such that the height range extends from the height at which it explodes 90% of the time to that at which it explodes 10% of the time. This method gives good results in that it reports per cent explosion as a function of drop height and reproduction of results is fairly good. The difficulty in the above method is that it is long and tedious and because of this few comparative results are available.

Another and more practical approach to the problem is the method used at the Naval Ordnance Test Station. This method, which was similar to the "Bruceton" method, consists of a series of tests, perhaps 20, in which the sequence of tests is carried out in a precise manner. Here, a weight is dropped upon a fixed quantity of explosive and if an explosion occurs, the weight is dropped from a height a fixed distance lower for the next test. Thus the impact is changed for every test and in a precise and organized manner. Statistical methods are used to interpret the the test results.

The "Naval Ordnance Test Station" method also specifies the handling of variables, such as the ratio of hammer weight to sample weight and the nature of the impacting surfaces. In general, this method seems the most practical and was adopted with certain modifications for this investigation.

Because of the unreliability of impact sensitivity results, few investigations have been carried out on anything but pure explosives and melted mixtures thereof. Explosives which were either immiscible or not practical to melt have not been well studied.

Trinitrobenzene (TNB) and trinitrotoluene (TNT) are examples of explosives which are not practical to melt together because, although their chemical structures are very similar, differing only by one methyl group, their melting points and other properties differ considerably. For this investigation it was necessary to mix them together physically to study the impact sensitivity of various mixtures.

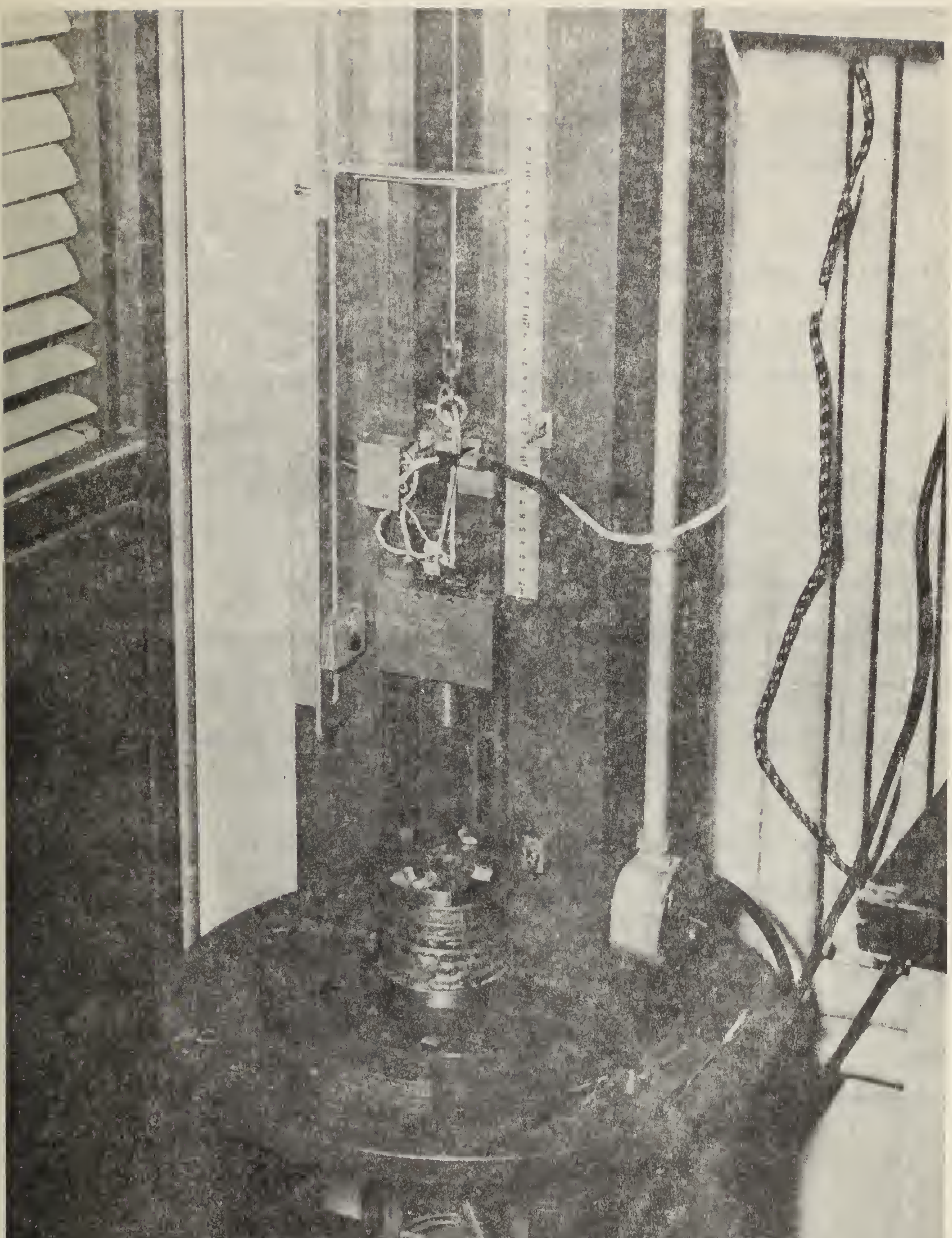
Several months after the above tests, additional work was done by Robinson and Small, using the same apparatus and techniques, on mixtures of TNT and PETN (Pentaerythritol Tetranitrate). This work is included in this report.

It is hoped that these results will add to the general knowledge of explosives and perhaps throw some light upon the problem of impact sensitivity testing.

An impact tester is essentially a machine which drops a fixed weight through a fixed distance. The important consideration is the production of reproducible impacts. There are many different types: some drop a ball shaped weight, some drop a weight that is on a hinged arm, and some drop along a vertical track. The machine in which the hammer drops along a vertical track seems to be the most rugged and is used in this project.

The impacter (See Fig. 1) is a steel tower with a geared motor at the top which raises and lowers a carriage containing two magnets. One magnet holds the impacter hammer so that there is no mechanical connection to hinder its free fall. The other magnet operates a pin that prevents the hammer from falling accidentally.

The hammer slides on lubricated vertical tracks with practically no friction. Electrical switches are operated automatically so that the hammer rises to a preset height, falls and is picked up again by the throwing of one switch. The sliding hammer falls upon a steel cylinder known as a "floating" hammer and weighs approximately 85 grams. The floating hammer rests upon the explosive being tested. The explosive rests upon a half inch square of special garnet paper which is on a steel anvil. The assembly is contained in a steel cup which like the entire machine is removable. The cup is enclosed by a steel cylinder called the "nest". The "nest" is warmed to 100°F



by brazed copper coils fed with water from a thermally regulated bath in the base of the impacter. The cup and the nest contain a hole in their respective bottoms through which an externally operated plunger can raise or lower the anvil to load and unload the charge. (See Fig. 2).

The advantages of the confinement of the explosive in the steel cup are: (1) Temperature control is more easily achieved, and (2) No special safety precautions are necessary beyond wearing a face shield.

The amount of explosive material used for a test is conveniently metered by means of a commercial powder measure which is mounted near the impacter. Since the powder measure delivers by volume, it must be calibrated for each different explosive or mixture. The measure is designed for larger volumes than needed for a single series of tests so a molded polyethylene plug is used to occupy the excess volume.

The mixtures of explosives are prepared by grinding, screening, and dry mixing. For the grinding operation, a standard Ro-Tap machine was used. The particle size of the explosive was determined by standard "Tyler" screens, and the dry mixing was done by a "Fisher Kendall" mixer. (See Appendix).

With the exception of the impacter, all equipment used was standard laboratory gear.

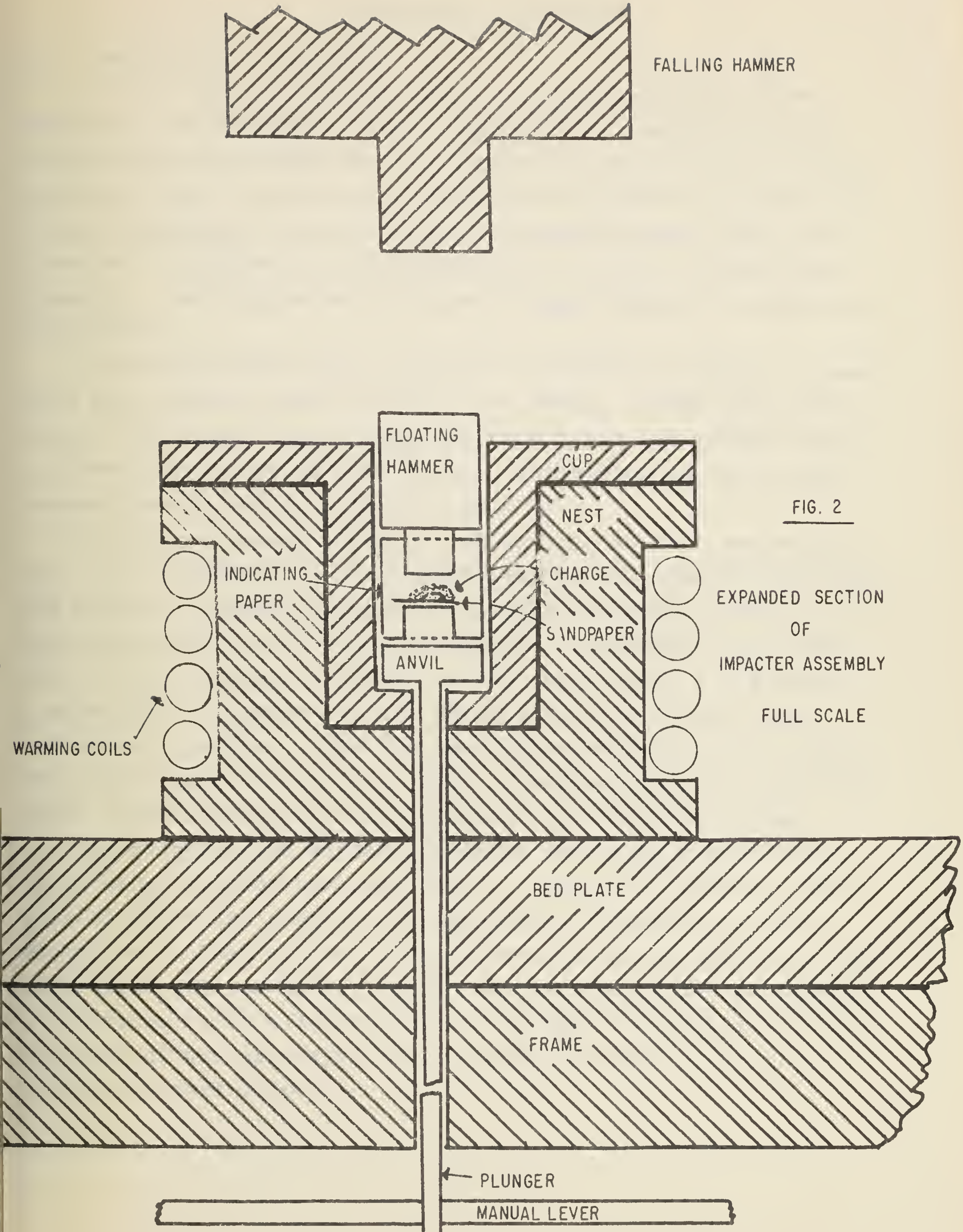



FIG. 2

EXPANDED SECTION OF IMPACTER ASSEMBLY FULL SCALE

1. Preparation of Samples

1, 3, 5-trinitrobenzene and 2,4,6-trinitrotoluene were obtained from "Eastman Organic Chemicals". Pentaerythritol Tetranitrate was prepared in the organic laboratory by officer students under the direction of Professor McFarlin of the U.S. Naval Postgraduate School. Chalk, prepared powder USP, was obtained from Eimer and Amend Division of Fisher Scientific Company. The samples were prepared by grinding in the following manner.

The pure material was placed on a number 70 "Tyler" screen with five copper discs the size of a penny. Underneath was a number 120 screen also containing five discs and beneath that a number 140 screen with no discs. The bottom of the screen assembly was a solid or "thru" pan.

The screen assembly was placed in the "Ro-Tap" and shaken until all the material had passed through the number 120 screen. The material passing through the number 140 screen was rejected. Therefore the size of the particles of the material used was small enough to pass through the 120 screen with a 125 micron opening but too large to pass through the 140 screen with a 105 micron opening. This size was selected because of screening convenience and also for ease of mixing since the particles would "flow" easily in the "tumbling" operation of mixing.

The grinding and sizing procedure used here was selected after other methods had been tried. The use of either a ball mill or motar and pestle with subsequent screening is quite dangerous in that clouds of fine dust are raised which are not only explosive but highly toxic both internally and externally. In addition, a considerable static charge is accumulated by the movement of the explosive in non-metallic media so that a sparking hazard exists.

The grinding on the brass and copper screens in the "Ro-Tap" still raised dust and accumulated a static charge but after a few minutes the dust settled and the charge disappeared due to the conducting nature of the apparatus. In this manner, the finely divided material was handled the minimum number of times both safely and easily.

In tests involving the pure material no further preparation was used. Whenever it was desired to mix materials, the powders were weighed on an analytical balance in the proper proportion; placed in vials with ground glass stoppers and mixed in a "Fisher Kendall" mixer. The action involved is a continuous rotation of the container with a simultaneous rocking motion through an angle of ninety degrees, which, according to the manufacturer, should give a uniform distribution in five minutes mixing time. Since mixing time did not hinder other phases of the project, all samples were mixed for 30 minutes and no lack of uniformity was detected. After mixing, the samples were allowed to stand for twenty-four hours to allow them to discharge any accumulated static charge.

2. Testing Procedure

The prepared samples were placed in an "Ideal" powder measure which was adjusted to "throw" the desired amount of material. The measure was adjusted until the amount thrown was within plus or minus one milligram.

The measured sample fell from the powder measure upon the rough surface of a half inch square of 180 grit, 5WD43 grade, garnet paper manufactured by the Carborumdum Company, in a rough cone shape. The garnet paper containing the coned material was placed on the anvil (See Fig. 2), which was raised from the cup to receive it, with the explosive side upward. The anvil containing the charge was lowered into the cup and a strip of white paper, long enough to go all the way around the cup and approximately one half inch in width, was placed in the cup. The floating hammer was then placed in the cup.

The floating hammer pushed the paper down until it formed a ring around the cup, one half inch from the charge. Another action of the floating hammer was to flatten the charge until a circle approximately three eighths of an inch in diameter was formed. The size of the circle was reasonably uniform as to diameter and varied with the size of the charge in thickness only. The meter stick which measured the drop height was adjusted for each series of tests because the thickness of the sample varied, and the impacting surfaces were ground smooth for each run of approximately 25 individual tests, thereby making adjustment necessary for each run.

After each test, the floating hammer was lifted manually and the white paper examined. If the charge had fired, the paper was blackened and this was used as the criterion of explosion. This technique was found to be necessary as other indications were often misleading. At first the criterion of a blackening of the sandpaper was used. When results were obtained which seemed improbable, sugar was substituted for the explosive. The sandpaper was blackened by the sugar when impacted. Since sugar does not explode under these conditions, this effect necessitated a change in the method of detection.

The use of paper to detect explosions in this method still requires care and skill, for in many cases bits of garnet paper are pushed through the white indicating paper which is subsequently discolored without any evidence of explosion. Therefore some judgment must be exercised in examining the paper.

The falling hammer was dropped automatically from pre-set heights on the floating hammer. In the first series of runs, a five pound falling hammer was used. The ratio of hammer weight to sample weight, which is two kilograms to twenty milligrams, was felt to be inapplicable here since sample weight varied from six to sixty milligrams. In subsequent runs the amount of charge was fixed to 23 milligrams so that the falling hammer was adjusted to 2.3 kilograms.

Following each impact or test, the floating hammer was lifted from the cup and placed on the side of the next, to keep the hammer from cooling. Then the paper was examined, a record of the test taken, and the height for the next impact test set. The anvil was raised from the cup, but not removed, scraped with a scapel and blown off with compressed air. The next sample was placed on the anvil, dropped down in the cup and the indicating paper put in place. Then the floating hammer was scraped, blown off, and placed in the cup.

After each run, the impacting surfaces of the anvil and floating hammer were sanded smooth. The falling hammer was checked for tightness, and the cup assembly washed with acetone to remove any unexploded material. In experiments of this type, a certain amount of unexploded material accumulates so that it is necessary to burn daily all fragments together with the acetone washings to avoid any excessive build-up of waste explosive. This was done in a large bucket in the fume hood with no difficulty.

## THE STATISTICAL APPROACH

In testing the impact sensitivity of an explosive, a hammer is dropped from a certain height on the sample under precisely controlled conditions. If no explosion occurs, the hammer is raised a definite distance higher and the test repeated. If an explosion occurs, the hammer is lowered the same distance and the test repeated. Normally this procedure is continued for twenty-five tests and the results analyzed.

This procedure assumes that the detection of explosion is certain and, as explained previously, it is not always certain. Even with the refinement of the indicating paper detector, some results are doubtful. Other uncertainties, such as the height at which to start the run, how many tests to make for one run, and when to stop a run, appear. Therefore certain arbitrary rules were set up and followed to make the tests meaningful.

The runs were all made under the following rules:

1. Every run was started at one hundred centimeters for the first drop height. If an explosion occurred, the drop height was moved down twenty centimeters, or if no explosion occurred the next drop height was twenty centimeters higher. This procedure was followed until the pattern changed. For example: explosions occurred at 100, 80 and 60 centimeters, but no explosion at 40. The next test would be at 45 centimeters, and the recording of the data would start. On the other hand, assume no explosion occurred at 100 and 120 centimeters, but

[illegible]

[illegible]

an explosion did occur at 140 centimers. The next test would be at 135 centimeters and the recording would start. This procedure was felt necessary because it established a definite method by which the explosive range could be found. When runs were repeated, this same technique was followed even though the range was purportedly known. The ranging shots however were not recorded, because since they were all performed in the same manner, the results could be obtained if necessary.

2. On those occasions, when it is difficult to judge whether or not an explosion has occurred, the decision is arbitrarily made that the result is the same as the previous test.

3. After twenty tests have been recorded, the run continues until the next change, either no explosion or explosion. This provides a rule for ending the run in a logical fashion.

4. The 50% point (FPP) as determined under these conditions, is used as the criterion in all impact tests.

These rules have been found to be helpful for they provide positive decisions in cases where some doubt may exist.

Another concept is necessary in interpreting the data. If an explosion occurred at 50 centimeters for a certain test, it is assumed that the explosion would also have occurred at any higher point since the impact would be greater. Also if no explosion occurred at 45 cm for another test, it is assumed that no explosion would occur at any lower point since the impact would be less. If the data are organized in this fashion

[illegible]

[illegible]

[illegible]

with drop heights vertically charted and the test numbers horizontally charted, this extra information obtained by these assumptions can be added and the percent probability for explosion at any height ascertained.

This method is illustrated in Figure 3 with "E" representing an actual explosion and "N" representing an actual no explosion. The assumed explosions are represented by "e" and the assumed no explosions by "n". From this illustration the height at which the per cent probability of explosion is 50% can be obtained. This can be seen to be 65 centimeters by plotting drop height versus per cent explosion. (See Fig. 4). The assumed points have aided the test results in that the number of tests considered is 79, instead of 22 and the assumed points are probably as accurate as the actual points.

As can be seen from Fig. 3, the data from the actual tests alone would give contradictory results so that in lieu of a great many tests, some device such as the assumed point concept should be adopted.

An additional refinement is used in this report. The per-cent probability is plotted versus drop height on probability paper instead of normal rectangular graph paper. This enables one to draw the best straight line through the plotted points and obtain the 50% point, assuming normal probability.

In evaluating the results from these data, it was observed that some runs seemed to be more reliable than others. This reliability or reproducibility (RSP) is believed to be a

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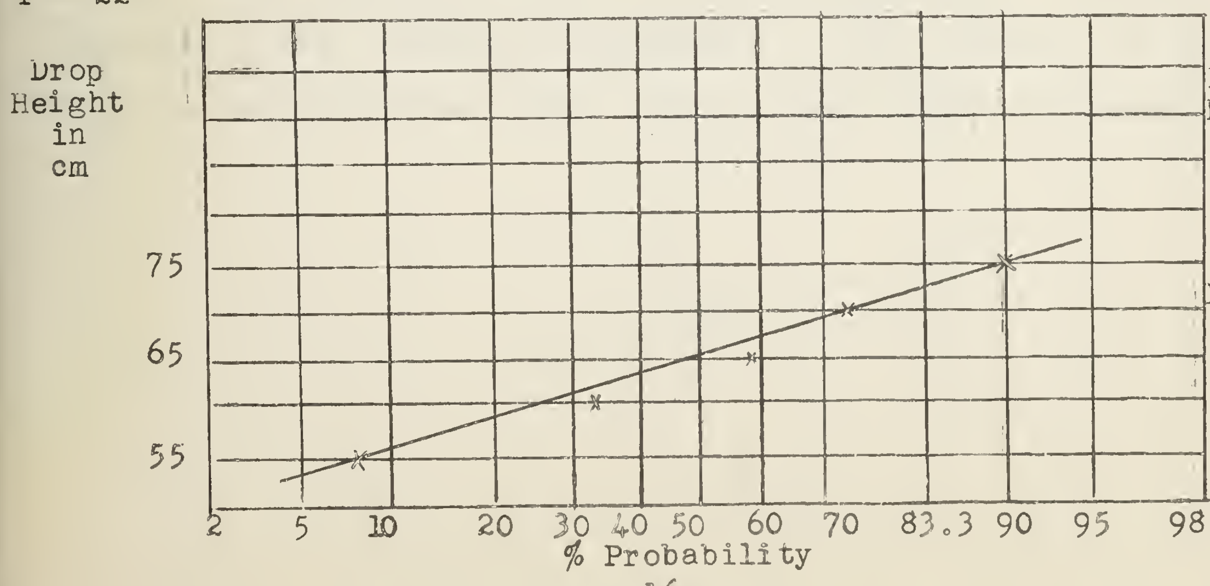
Explosives Laboratory

Impact Test Data Sheet

| 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | 16 | 17 | 18 | 19 | 20 | 21 | 22 | 23 | 24 | 25 | X Ratio | % | Remarks |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | | | | | | | | | | | | | | | | | | 12/29/55 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | TNT Powd |
| | | | | | | | | | | | | | | | | | | | | | | | | | | 33 mg.chg |
| | | | e | e | e | e | | e | | | e | e | e | | | | | | | E | | | | 10/10 | 100 | |
| | | | E | e | e | e | | e | | | e | e | e | | | | | | N | | | | | 9/10 | 90 | |
| | | N | | E | e | e | | e | | | e | e | e | | | | | N | n | | | | | 8/11 | 72.8 | |
| | N | n | | | E | e | | e | | | E | e | e | | | | N | n | n | | | | | 7/12 | 58.2 | |
| N | n | n | | | | E | | E | | N | | E | e | | | N | n | n | n | | | | | 4/12 | 33.3 | |
| n | n | n | | | | | N | | N | n | | | E | | N | n | n | n | n | | | | | 1/12 | 8.3 | |
| n | n | n | | | | | n | | n | n | | | | N | n | n | n | n | n | | | | | 0/12 | 0 | |

N = 7 $\quad RSP = 1 - \frac{N-2}{T-1} \qquad RSP = 1 - \frac{5}{21} = .762$

T = 22




FPP = 65
RSP = .76

Fig.3

function of the explosive itself under impact and has no direct relationship to the FPP.

The following formula was suggested in an effort to assign a numerical value to the RSP.

$$RSP = 1 - \frac{n - 2}{T - 1}$$

n = the number of 5 cm increments for 100% E to 0% E

t = the number of actual tests within the above limits.

This formula would then assign the value of 1 to the perfect explosive and the value of 0 the worst explosive that would meet the rules set up in the beginning of the chapter.

Figure 3 illustrates the use of the formula. Here N = 7 and T = 22. Therefore the RSP = .76. If N had been greater, this would have indicated a greater spread between the 100% and 0% explosion points, and the RSP would have been lower. Conversely, a smaller spread would result in a higher RSP.

Occasionally runs are made in which tests fall either above or below the .0 to 100% range. These tests provide very little information since the tests above the range are all explosions and the tests below the range are all no explosion. Therefore these tests are not used in determing T. This lowers the RSP value because there are fewer tests in the significant region.

## EXPERIMENTAL RESULTS

Before comparing the impact sensitivities of explosive mixtures an investigation was made upon the sensitivity of varying amounts of material. The discovery was made that there is a most sensitive amount of an explosive. This has not been reported in the literature and is considered to be an important contribution.

The most sensitive amount of both TNB and TNT is approximately $23 \pm 1$ milligrams. The lower amounts are less sensitive because the explosive layer is thinner and since it is spread on garnet paper, the garnet grains are large enough to absorb an appreciable part of the shock. The greater amounts are also less sensitive, but this is due to a dilution factor. The impact is divided among more particles of explosive, making less the probability of any one particle absorbing enough energy to explode.

This could perhaps be thought of as the energy to cause explosion by impact expressed in calories per mol. This energy for pure TNT is $30 \pm .5$ kilocalories per gram mol as computed for the 50% point and $34.5 \pm$ kilocalories/gm mol for the 100% point assuming the explosive absorbed the entire impact. The energy necessary for the initiation of TNT, as calculated from curves in reference 4, was 38.3 kilocalories/gm mole. The fact that these results are of the same order of magnitude is interesting because one was computed from impact results and the other from temperature-time data. However one should be cautious in attributing any significance to this observation.

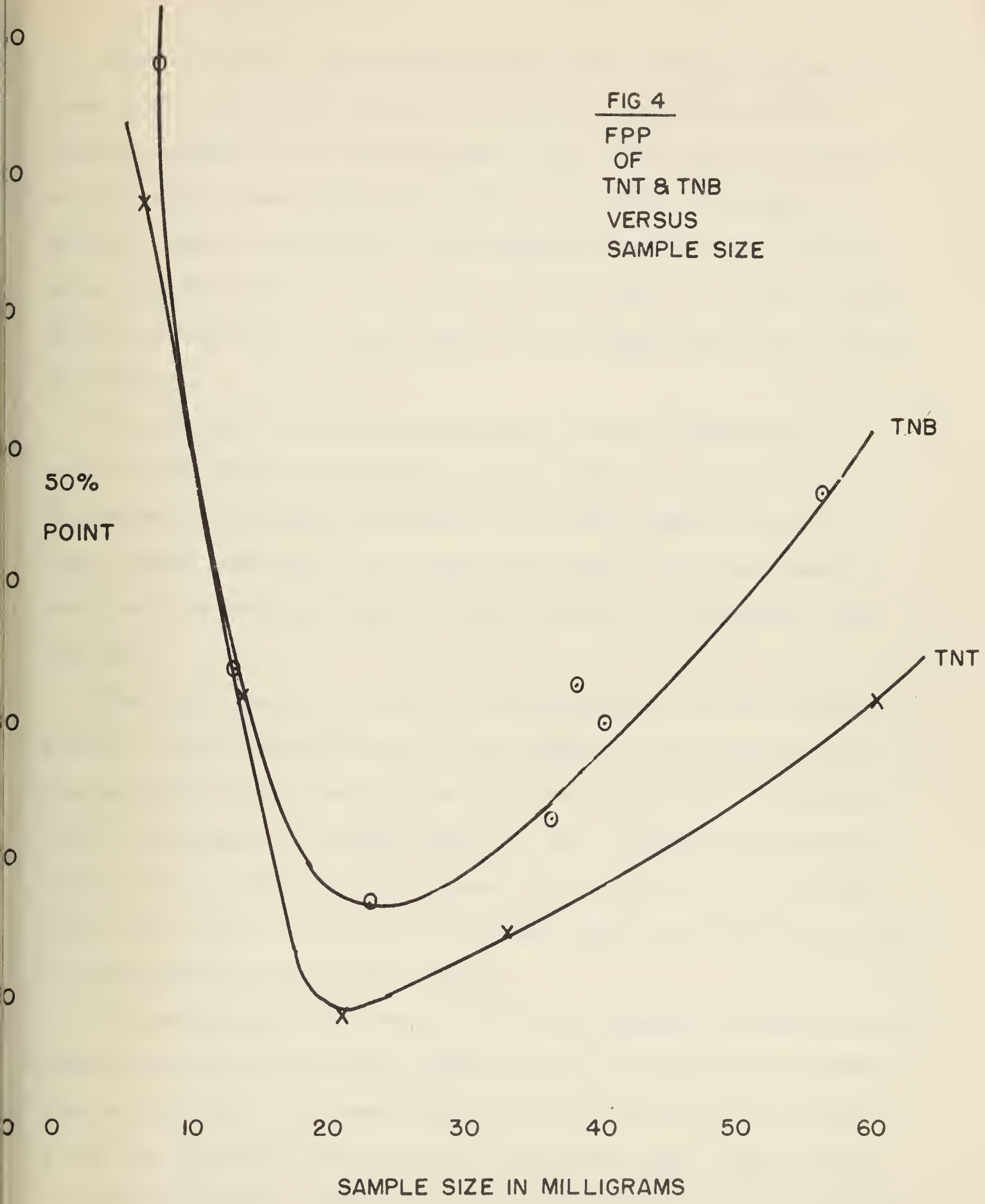


FIG 4
FPP
OF
TNT & TNB
VERSUS
SAMPLE SIZE
TNB
TNT
50%
POINT
0
10
20
30
40
50
60
SAMPLE SIZE IN MILLIGRAMS

After the most sensitive amounts of the two explosives had been found, (See Fig. 4), the influence of large amounts of foreign material was investigated. Runs were made on mixtures of chalk with each explosive. In these tests, the amount of mixture used for each test was determined by the most sensitive amount of material found in the previous series of runs. This was 23 milligrams, so the tests were all made using this amount of mixture.

The results from the second series of runs (See Fig. 5), were of the nature of dilution alone, that is the sensitivity decreased in a reasonable manner up to the highest point of drop height available. An interesting facet of these results was that the TNB apparently is more affected by dilution than the TNT.

The third series of runs in which TNB and TNT were mixed and the mixture sensitivity studied show an interesting trend. The sensitivity of the mixture is above that of the TNT until 75% of the material is TNB (See Fig. 6). In no case did the sensitivity of the mixture become lower than that of the TNB. This shows that the effect of the TNB upon the TNT and vice versa is considerably more than dilution.

To investigate this effect, Robinson and Small repeated the third series of runs using pentaerythritol Tetranitrate (PETN) instead of TNB. The same techniques and apparatus were used as in the previous run, as well as the same TNT. This result is plotted on Fig. 6 and the curve is virtually a straight line.

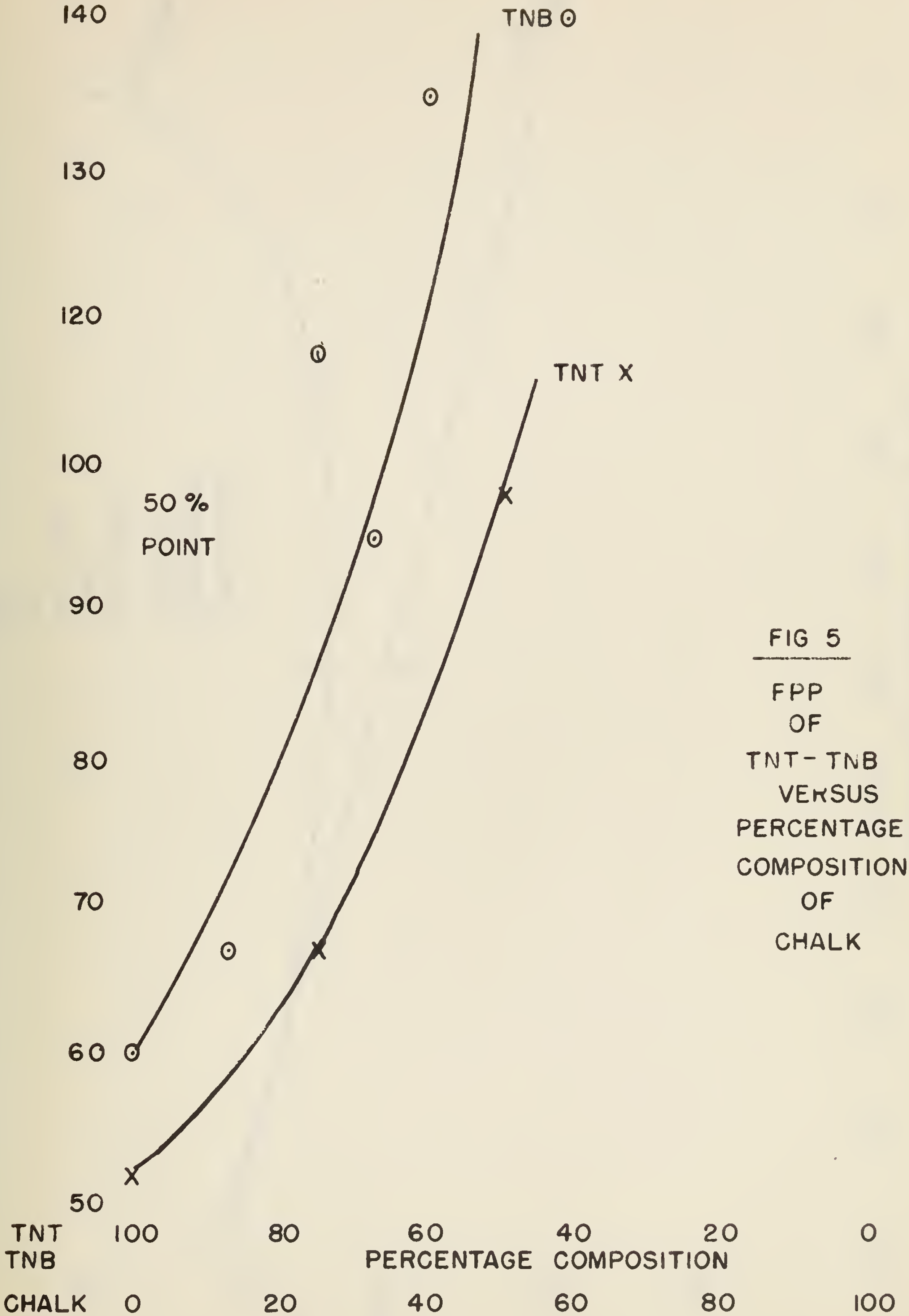



FIG 5

FPP
OF
TNT - TNB
VERSUS
PERCENTAGE
COMPOSITION
OF
CHALK

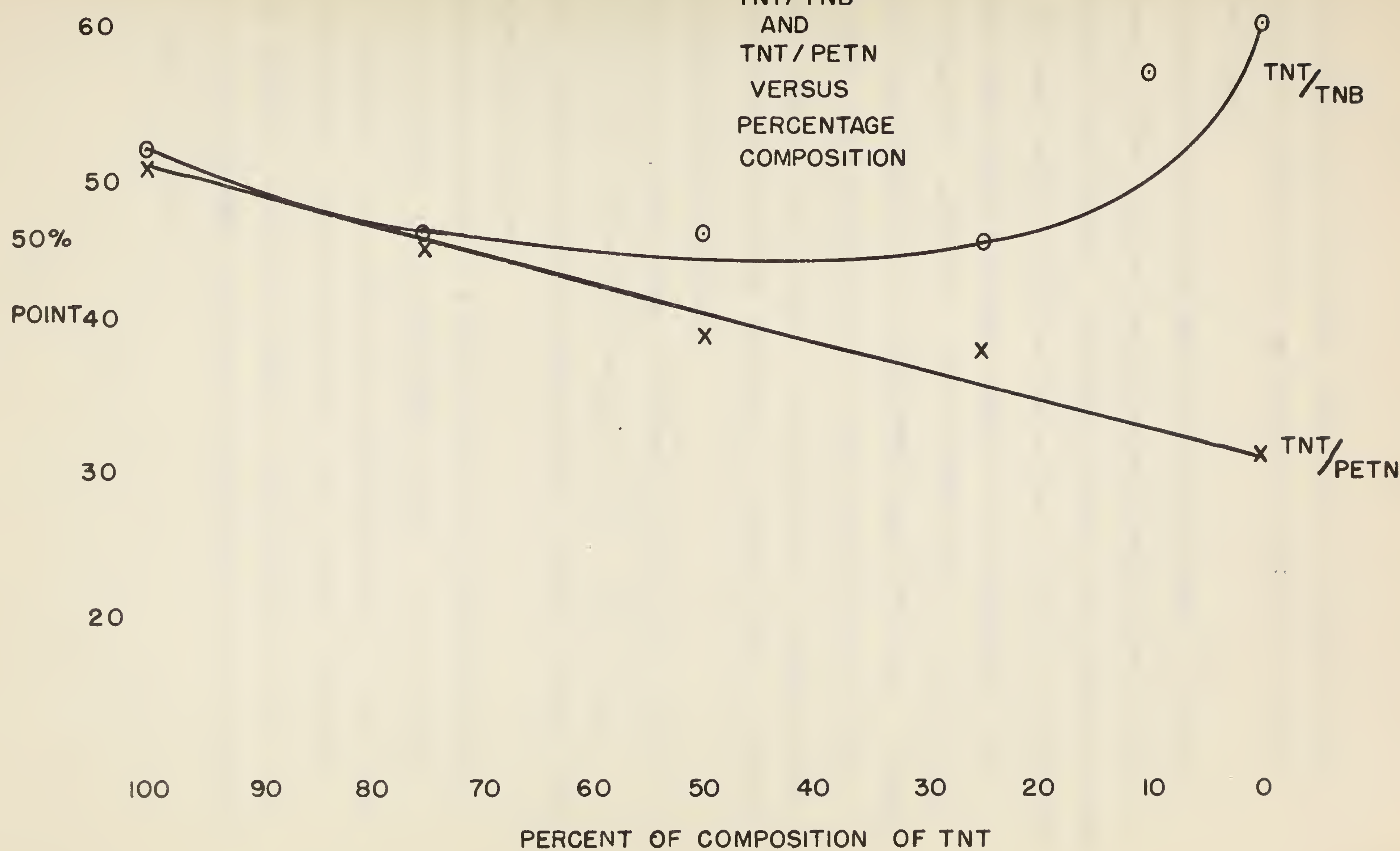



FIG 6
FPP OF TNT/TNB AND TNT/PETN VERSUS PERCENTAGE COMPOSITION

It was gratifying to discover that both the FPP and the RSP were reasonable checks of the earlier run in spite of a lapse of several months.

The properties of the RSP were also investigated. In Fig. 7, the RSP was plotted against the percentage composition of both of the mixtures being studied. Peculiarly enough, the curves obtained are of the same general shape as the curves of Fig.6. The TNT-TNB versus RSP curve shows a minimum similar to the minimum of the TNT-TNB versus FPP curve while the TNT-PETN curve is essentially a straight line similar to the TNT-PETN versus FPP curve. However both of these comparisons suffer in that the respective curves are mirror images of one another. Also there are wayward points that do not follow the above reasoning. Further work is planned to investigate these objections.

Another set of curves (Fig.8) was made plotting the size of the charge of both TNT and TNB versus the RSP. Both curves indicate a greater reliability factor with increasing sample size which seems quite logical.

Therefore some change has taken place in the TNT-TNB mixture that has not occurred in the TNT-PETN mixture. It is probable that the lowering of the FPP in the TNT-PETN mixture is due to a mutual solubility of the two materials under impact conditions since the shape of the curve suggests a melting point curve. In the case of the TNT-PETN, structural dissimilarity would probably prevent mutual solution.

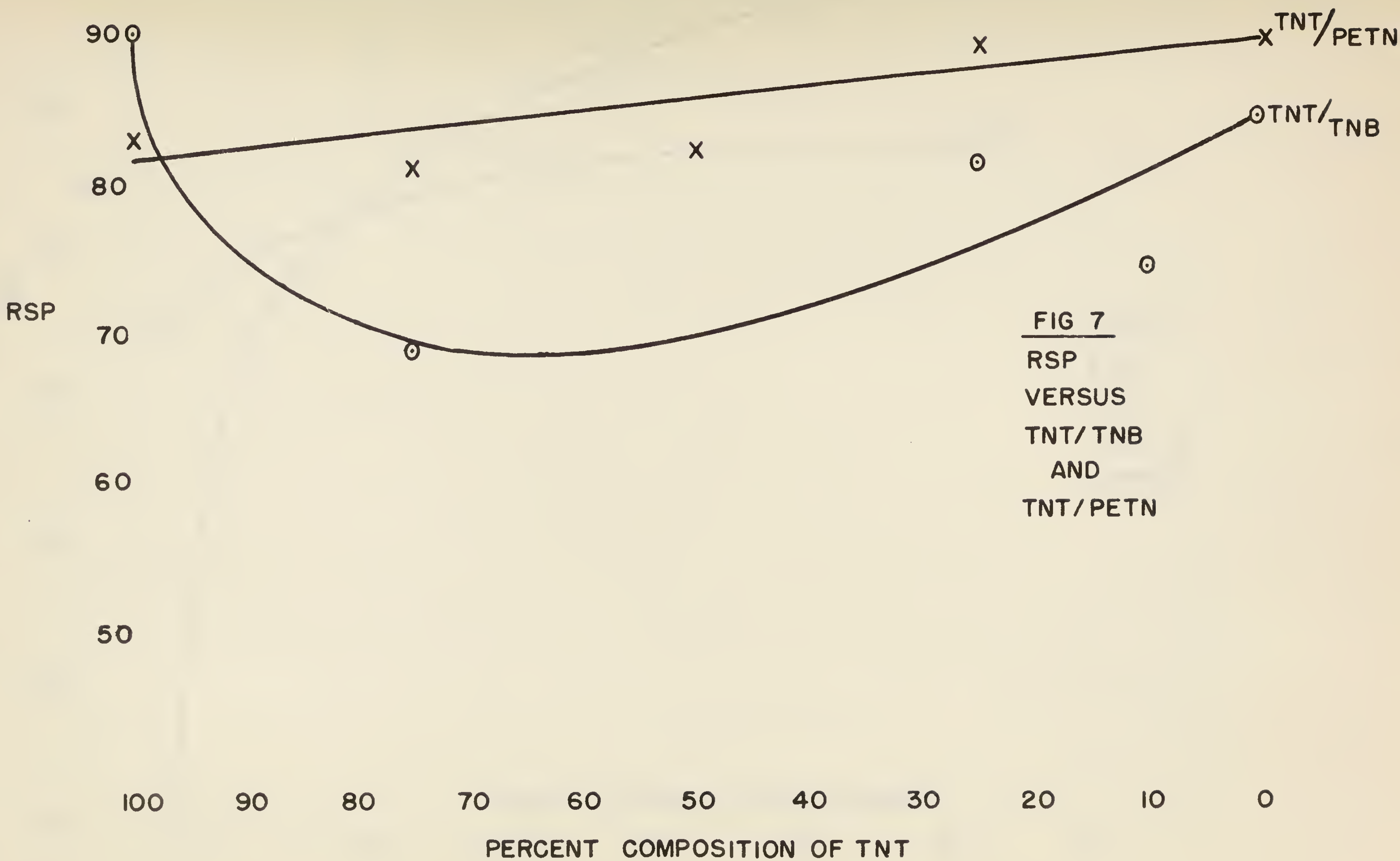
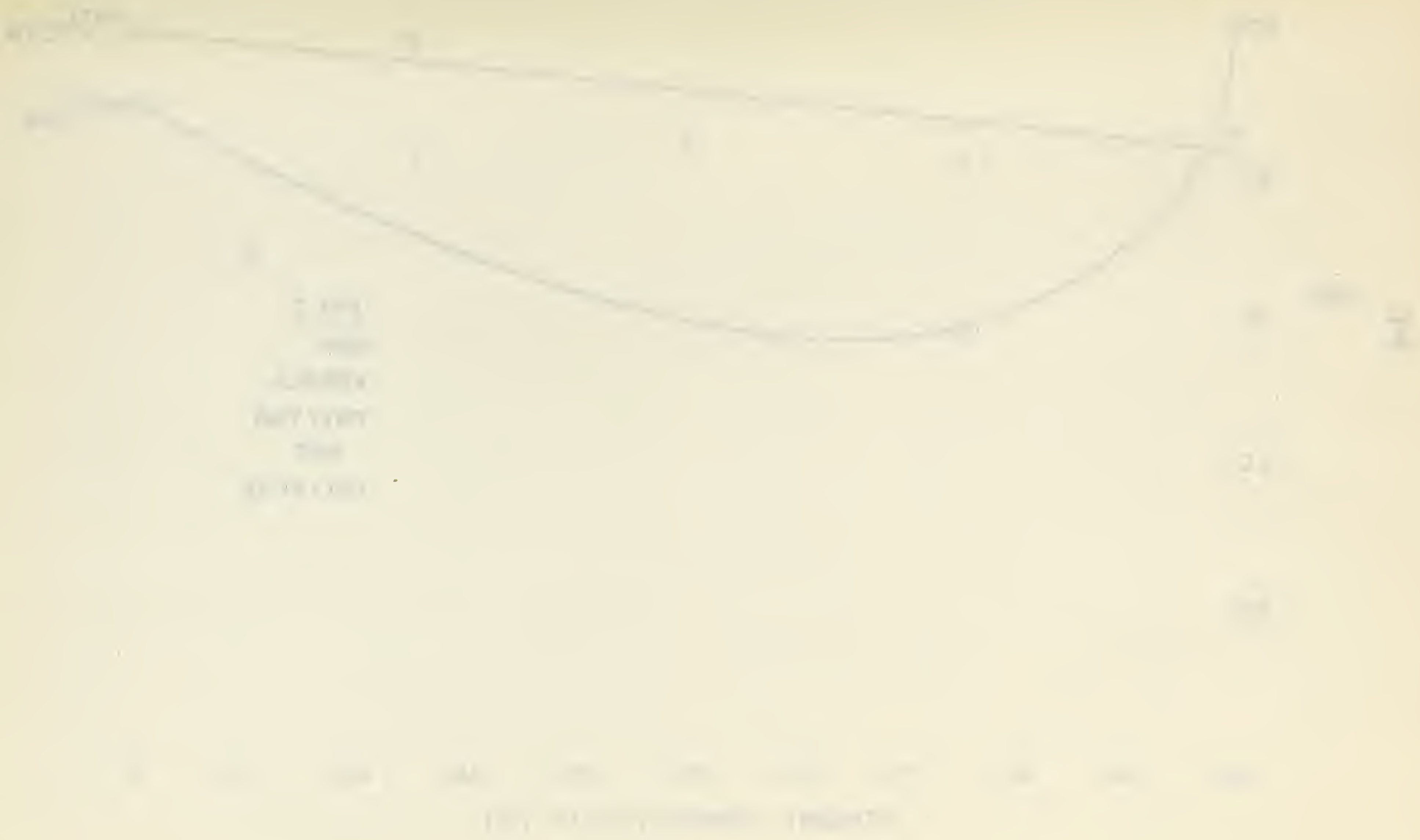



FIG 7

RSP VERSUS TNT/TNB AND TNT/PETN



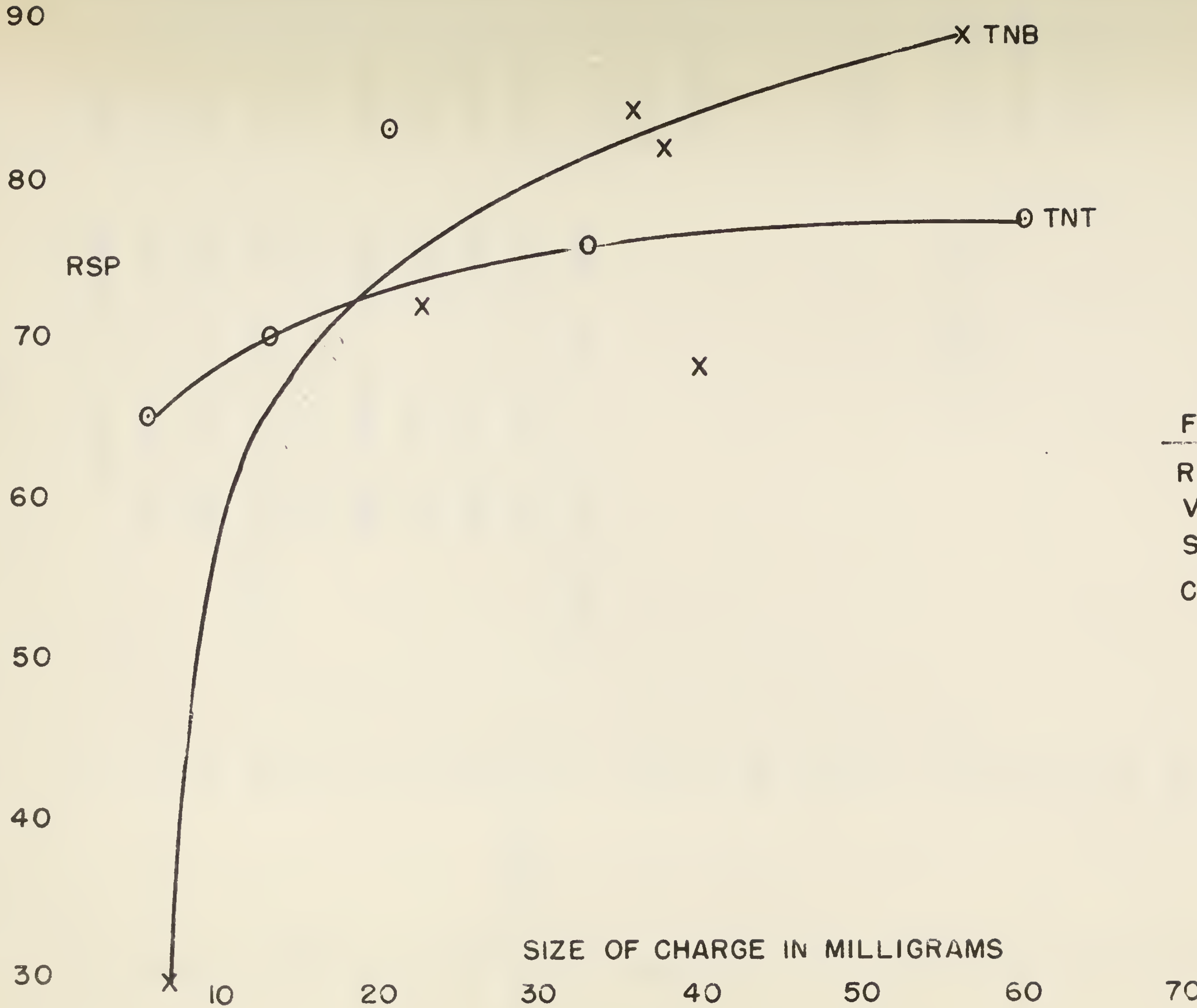



FIG 8

RSP
VS
SIZE OF
CHARGE

## SUMMARY CHART OF PROJECT

| No. | Date | Sample | Size of Change in mg | FPP in cm | RSP |
|---|---|---|---|---|---|
| | 12/29/55 | TNT (Batch 1) | 6 | 118 | .65 |
| | " | " | 13.5 | 82 | .70 |
| | " | " | 33 | 65 | .76 |
| | 1/4/56 | " | 60 | 82 | .77 |
| | 3/13/56 | " | 21 | 59 | .83 |
| | 1/4/56 | TNB (Batch 1) | 13 | 84 | .65 |
| | " | " | 7 | 128 | .35 |
| | 1/11/56 | " | 36 | 73 | .84 |
| | " | " | 56 | 97 | .89 |
| | " | " | 38 | 83 | .82 |
| | 1/16/56 | " | 40 | 80 | .68 |
| | 1/18/56 | " | 23 | 67 | .72 |
| | 3/12/56 | TNB (Batch 2) T-120 On 140 | 23 | 60 | .84 |
| | 3/14/56 | TNB 50% - Chalk 50% | 23 | No expl. to 150 cm | |
| | 3/14/56 | TNB 75% - Chalk 25% | 23 | 115 | .65 |
| | 3/15/56 | TNB 87% - Chalk 13% | 23 | 67 | .50 |
| | 3/15/56 | TNB 67% - Chalk 33% | 23 | 95 | .82 |
| | 3/20/56 | TNB 60% - Chalk 40% | 23 | 135 | .44 |
| | 3/15/56 | TNT (Batch 2) T-120 On 140 | 23 | 52 | .90 |
| | 3/16/56 | TNT 50% - Chalk 50% | 23 | 98 | .68 |
| | 3/20/56 | TNT 75% - Chalk 25% | 23 | 67 | .94 |
| | 3/28/56 | TNT 50% - TNB 50% | 23 | 46 | .82 |

SUMMARY CHART OF PROJECT

(Continued)

| No. | Date | Sample | Size of Change in mg | FPP in cm | RSP |
|---|---|---|---|---|---|
| 3 | 3/29/56 | TNT 25% TNB 75% | 23 | 45 | .81 |
| 4 | 3/29/56 | TNT 75% TNB 25% | 23 | 46 | .69 |
| 5 | 3/30/56 | TNT 10% TNB 90% | 23 | 57 | .74 |
| 6 | Aug.'56 | TNT (Batch 2) T-120 On 140 | 23 | 51 | .83 |
| 7 | " | TNT 75% PETN 25% | 23 | 45 | .81 |
| 8 | Sept.'56 | TNT 50% PETN 50% | 23 | 39 | .82 |
| 9 | " | TNT 25% PETN 75% | 23 | 38 | .89 |
| 0 | Oct. '57 | PETN 100% | 23 | 31 | .90 |

[illegible]

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